

Effective Date: 10/15/01

STL On-Site Technologies STANDARD OPERATING PROCEDURE

TITLE : Semivolatile Organic Compounds (SVOCs) by GC/MS

SUPERSEDES : (NONE)

This documentation has been prepared by the On-Site Technologies (OST) Division of Severn Trent Laboratories (STL) solely for STL's own use and use of STL's customers in evaluating its qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to Severn Trent Laboratories upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF SEVERN TRENT LABORATORIES IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY SEVERN TRENT LABORATORIES IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

© COPYRIGHT 2001 SEVERN TRENT LABORATORIES. ALL RIGHTS RESERVED.

Approvals and Signatures

Division Manager:

Peter A. Law

Date

QA Manager:

Robert S. DeGaetano

Date

Technical Director:

John V. Hawkins

Date

Effective Date: 10/15/01

1.0 SCOPE AND APPLICATION

- 1.1 The purpose of this SOP is to outline the techniques for determining the presence and concentration of various semivolatile organic target and non-target compounds. The standard 8270C target compounds are listed in Table 1.0. The method used in this procedure is solvent extraction and gas chromatograph/mass spectrometer analysis.
- 1.2 It is the policy of STL and of the On-Site Technologies Division to ensure that we administer contracts and orders for goods and services in a manner that is fully compliant with governmental laws and regulations, as well as the STL Policy Statement on Business Ethics and Conduct.

2.0 DEFINITIONS

- 2.1 There are many definitions used within the laboratory that may be generic to all laboratory analyses or more specific for certain methods. For the most recent terms and definitions used within the laboratory, refer to the STL-OST Laboratory Quality Manual.

3.0 SUMMARY OF METHOD

- 3.1 This method employs the technique of solvent extraction, coupled with a gas chromatograph/mass spectrometer analysis. An aliquot of sample extract is injected onto a gas chromatograph. The fused silica capillary column is then temperature programmed to separate the semi-volatiles organics prior to detection by the mass spectrometer.
- 3.2 This method is based on SW-846 Method 8270C and Method 8000B. The concentration of the solution used for the initial demonstration of precision and accuracy varies from that listed in the method. The lab uses a concentration of 20 ug/l , and the concentration listed in Table 6 of the method is 100 ug/l. Table 9 of this SOP reflects that change. When linear regression is used for quantitation the method calls for the use of percent drift to verify the validity of the continuing calibration. Because of software limitations the lab will use percent difference instead. The lab will use the criteria listed in the method.

4.0 INTERFERENCES

- 4.1 Method interferences may be caused by contaminants in solvents, reagents, and laboratory solvent vapors. This can lead to discrete artifacts and/or elevated baseline in the gas chromatograph. All these materials must be demonstrated to be free from interferences by the running of laboratory reagent blanks.

Effective Date: 10/15/01

5.0 SAFETY

- 5.1 Procedures shall be carried out in a manner that protects the health and safety of all STL-OST associates.
- 5.2 The STL Chemical Hygiene Plan (CHP) gives details about the specific health and safety practices which are to be followed in the laboratory area. Personnel should receive training in the CHP, including the written Hazard Communication plan, prior to working in the laboratory.
- 5.3 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined, therefore each chemical compound should be treated as a potential health hazard. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:
 - 5.3.1 Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include: Acrylonitrile, benzene, carbon tetrachloride, chloroform, 1,2-dibromo-3-chloropropane, 1,4-dichlorobenzene, and vinyl chloride.
 - 5.3.2 Chemicals known to be flammable are: Methanol.
- 5.4 Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.
- 5.5 The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operations will permit.
- 5.6 All work must be stopped in the event of a known or potential compromise to the health and safety of an STL-OST associate. The situation must be reported immediately to a laboratory supervisor.

6.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 6.1 All sample extracts for semivolatile analysis must be protected from light and refrigerated at 4°C from the time of extraction until analysis.
- 6.2 All sample extracts for semivolatile analysis shall be analyzed within 40 days of following extraction.

Effective Date: 10/15/01

7.0 APPARATUS AND MATERIALS

7.1 GC/MS/DS System

- 7.1.1 Gas Chromatograph - Hewlett-Packard Model 5890 GC, an analytical system which is temperature programmable with splitless injection and all required accessories including syringes, analytical columns, and gases.
- 7.1.2 GC Column - 30 m x 0.25 mm ID x 0.5 um film thickness RTX semivolatile column by Restek Corporation, or equivalent.
- 7.1.3 Mass Spectrometer - Hewlett-Packard 5970, capable of scanning from 35 to 500 amu every 1 second or less, utilizing 70 volts (nominal) electron energy in the EI ionization mode, and producing a mass spectrum which meets all the instrument performance criteria when 50 ng or less of DFTPP is injected through the GC inlet. Refer to Sections 10.3.1 for the performance criteria. Any samples analyzed when DFTPP criteria have not been met will require reanalysis.
- 7.1.4 GC/MS interface - any GC to MS interface that gives acceptable calibration points, at 50 ng or less per injection, for each of the parameters of interest, and achieves all acceptable performance criteria, may be used. GC to MS interfaces constructed of all-glass or glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane.
- 7.1.5 Data system -Hewlett Packard DOS Chemstation with Enviroquant Software, capable of continuous acquisition and storage, on machine readable media, of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that allows searching any data file for ions of a specified mass and plotting such ion abundances versus time or scan number (EICP). Software also allows integrating the abundance in any EICP between specified time limits. Also, software allows for the comparison of sample non-target spectrum against reference library spectra. The most recent release of the NIST/EPA/NIH mass spectral library shall be used as the reference library. The DOS data systems will flag all manual edits with "M" qualifier.
- 7.1.6 Syringes -various volumes.
- 7.1.7 Micropipets - various volumes.

Effective Date: 10/15/01

8.0 REAGENTS AND STANDARDS

- 8.1 Stock Standards - certified standards purchased from commercial sources in a semivolatile organics kit containing ampulated mixes of target compounds, matrix spike compounds, surrogates, and internal standards.
 - 8.1.1 New ampules are opened every 12 months, or sooner, if the standard has degraded or evaporated.
- 8.2 Working Standards
 - 8.2.1 Calibration Standard Solutions
 - 8.2.2 Calibration standards at six concentration levels (10, 20, 50, 75, 100 and 200 total ng/ul) are prepared from the stock solutions. Each standard contains all the target compounds and surrogates.
 - 8.2.3 Internal Standard (IS) Spiking Solution
 - 8.2.4 A 400 ng/ul IS spiking solution containing the internal standards 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12, is prepared from stock solutions every 12 months, or sooner, if the solution has degraded or evaporated. Add 10 ul of this solution to a 100 ul aliquot of extract for a concentration of 40 ng/ul (80 ng/2ul).
 - 8.2.5 Surrogate (SU), Matrix Spike (MS), and Full Matrix Spike (FMS) Spiking Solution
 - 8.2.6 The acid and base-neutral surrogate, matrix spike, and full matrix spike spiking solutions containing the appropriate compounds are prepared in methanol.

Effective Date: 10/15/01

9.0 PROCEDURES

9.1 Instrumental Conditions

Gas Chromatograph

Carrier Gas: Helium
Flow Rate: 30 cm/sec
Initial Temp.: 40°C
Initial Hold: 4 min
Ramp Rate 1: 10°/min
Second Temp.: 270°C
Second Hold: 12 min
Ramp Rate 2: 8°/min
Final Temp.: 310°C
Final Hold: 10 min
Injector Temp.: 265°C
Transfer Temp: 310°C
Injector: Grob-type, splitless
Sample Volume: 1 or 2 ul
EPC/EPP 99 mls/min to 40 psi for 0.3 min then 99 mls/min to 7.1 psi

9.2 Mass Spectrometer

Electron Energy: 70 eV
Mass Range: 35 - 500 amu
Scan Time: less than 1 sec/scan

9.2.1 The mass spectrometer must be tuned to meet the instrument performance check criteria for 50 ng or less of DFTPP listed in Table 4.0.

9.3 Calibration Standards

9.3.1 Six initial calibration standards containing all the semivolatile target compounds and SU's are analyzed by injecting 1 or 2 ul of the calibration standards prepared in section 8.2.1. If a 2 ul injection is used, the resulting in final concentrations are 20, 40, 100, 150, 200, and 400 total ng/2 ul. The IS compounds are added to all calibration standards at a final concentration of 40 ng/uL (80 ng/2ul).

9.3.2 The working calibration range of this method is defined by the initial calibration curve, 10 to 200 ng (20 ng to 400 ng for a 2 uL injection). All extracts with target compounds exceeding 200 ng/uL (400 total ng for a 2 uL injection) must be diluted to within the upper half of the calibration range.

Effective Date: 10/15/01

9.4 Initial Calibration

- 9.4.1 The calibration curve is prepared by adding the appropriate amount of each of the target analytes to an autosampler vial and adding 10 ul of Internal Standard Solution.
- 9.4.2 An initial calibration must be analyzed on each GC/MS system upon installation, whenever corrective action is taken which may affect the initial calibration criteria, or if the continuing calibration criteria can not be met.
- 9.4.3 The initial calibration can be performed only after the instrument performance is verified by meeting the DFTPP ion abundance criteria listed in Table 4.0. If less than 12 hours has expired since the tuning compound was injected, after meeting the initial calibration criteria, then samples may be analyzed. No continuing calibration is required. Quantitation is based on either the average RF in the curve or a linear regression line. If time does not remain in the 12 hour time period, a continuing calibration must be performed.
- 9.4.4 The response factors of the system performance check compounds (SPCC's) listed in Table 5.0 must have average relative response factors which are greater than or equal to 0.05, and the continuing calibration check compounds (CCC's) listed in Table 5.1 must have percent relative standard deviations (RSD's) less than or equal to 30.0 percent for the initial calibration to be acceptable.
- 9.4.5 There are three options that can be used to determine the acceptability of an initial calibration. They are listed below:
 - 9.4.5.1 If the %RSD of any compound is $\leq 15\%$, the average RF from the curve for that compound may be used for quantitation.
 - 9.4.5.2 If the %RSD for one or more analytes is greater than 15%, the mean %RSD for all compounds will be calculated. If the mean %RSD is $\leq 15\%$, the average RF may be used for all compounds.
 - 9.4.5.3 If the mean %RSD is greater than 15%, quantitation from the regression line must be used for those compounds whose %RSD is greater than 15%.

Effective Date: 10/15/01

9.5 Continuing Calibration

- 9.5.1 The continuing calibration can be performed only after the instrument performance is verified by meeting the DFTPP ion abundance criteria listed in Table 4.0. The calibration is verified by injecting a standard containing 50 ng/uL (100 total ng for a 2 uL injection) or less of DFTPP. The standard must be analyzed every 12 hours.
- 9.5.2 The response factors of the system performance check compounds (SPCC's) listed in Table 5.0 must have response factors greater than 0.05, and the continuing calibration check compounds (CCC's) listed in Table 5.1 must be less than or equal to 20.0 percent difference (%D) for the continuing calibration to be acceptable. The calculations are listed in Section 13.0. If the continuing calibration does not meet the above criteria the standard must be reinjected. If in the judgment of the analyst routine maintenance will solve the problem it can be performed prior to re-injection of the standard. Examples of routine maintenance include cutting the column, cleaning the injection port, and changing the injection port liner. Repeated failure may require corrective actions and reanalysis of the initial calibration.

9.6 Sample Analysis Procedures

- 9.6.1 Sample Extract Analysis
- 9.6.2 Sample extracts are removed from refrigerated storage and are signed out on the chain of custody form. All sample extracts are signed back in after they returned to storage.
- 9.6.3 Make sure all instrumental operating conditions are correctly set and DFTPP and calibration criteria have been met.
- 9.6.4 In an autosampler vial, load a measured aliquot of sample extract and spike with 10 ul of IS spiking solution.
- 9.6.5 The prepared extracts are then loaded into the HP autosampler carousel tray and the sequence is set up in the software to match the injection sequence on the autosampler tray.
- 9.6.6 If a sample is analyzed which contains target compounds at concentrations greater than the initial calibration upper limit, then the sample must be reanalyzed at an appropriate dilution.

Effective Date: 10/15/01

9.7 Qualitative Analysis - Target Compounds

- 9.7.1 The relative retention time of a target compound must be within +/- 0.06 RRT units of the RRT of the calibration standard for a positive identification. For reference the standard must be analyzed within the same 12 hour time period as the sample. If the sample is analyzed within the same 12 hour time period as the initial calibration, then use the 50 ng standard as the reference. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT shall be assigned by using the extracted ion current profiles for ions unique to the component of interest.
- 9.7.2 In addition, a comparison must be made between the mass spectrum obtained in the sample analysis and the reference mass spectrum for that compound, which was obtained on that specific GC/MS system. The requirements for qualitative verification by comparison of mass spectra are as follows:
- 9.7.3 All ions present in the reference spectrum at an intensity greater than 10% must be present in the sample spectrum.
- 9.7.4 The relative intensities of the ions above 10% must agree with 20% between the reference and sample spectra.
- 9.7.5 Ions greater than 10% in the sample spectrum but not present in the reference spectrum must be considered and accounted for by the analyst.
- 9.7.6 If a compound cannot be verified by the above criteria, but in the technical judgment of the analyst, the identification is correct, then the compound shall be reported.

9.8 Qualitative Analysis - Tentatively Identified Compounds

- 9.8.1 A library search may be performed upon request for non-target compounds in the sample for purposes of tentative identification. For this purpose, the most recent release of the NIST mass spectral library shall be used. Pesticide and PCB confirmation is also done by tentatively identified compound search if needed to prove the presence of these compounds from a GC analysis.

Effective Date: 10/15/01

- 9.8.2 Up to 20 non-target organic compounds of highest apparent concentration shall be tentatively identified via a forward library search. Only compounds with responses greater than 10% of the closest IS exhibiting no interference are to be searched. Peaks suspected of being aldol-condensation reaction products shall be searched and reported as such, but not counted as part of the 20 most intense non-target compounds. Solvent peaks which may be detected due to an early scan start time shall not be counted as a library search compound. All solvent peaks shall be labeled on the chromatogram as such.
- 9.8.3 A tentative identification will be made after a comparison between the mass spectrum obtained in the sample analysis and the library search mass spectra found for that compound. The requirements for tentative verification by comparison of mass spectra are as follows:
 - 9.8.4 Ions present in the reference spectrum at an intensity greater than 10% should be present in the sample spectrum.
 - 9.8.5 The relative intensities of the ions above 10% should agree with 20% between the reference and sample spectra.
 - 9.8.6 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or coeluting compounds.
 - 9.8.7 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible background subtraction by the data system.
 - 9.8.8 If in the technical judgment of the mass spectral interpretation specialist, no valid tentative identification can be made, the compound shall be reported as unknown. Additional classification shall be made if possible (i.e. unknown hydrocarbon).
- 9.9 Quantitative Analysis - Target Compounds
 - 9.9.1 Target compounds are quantitated by the internal standard technique. The associated internal standard used is listed in Table 6.0. The EICP area of the quantitation ions of compounds listed in Tables 7.0 and 8.0 are used.

Effective Date: 10/15/01

- 9.9.2 All manual integrations must be initialed and dated by the analyst. The average relative response factor (RRF) from the curve or the linear regression line from the curve is used to calculate the concentration in the sample. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion. If secondary ion quantitation is performed, document the reasons in the SDG narrative. The area of a secondary ion can not be substituted for the primary ion unless a RRF is calculated using the secondary ion.
- 9.9.3 When compound concentrations are below the EQL, but the compound meets identification criteria, report the concentration with a "J" qualifier.

Water Samples

$$\text{Concentration ug/L} = \frac{(A_x)(I_s)(V_t)(D_f)}{(A_{is})(RRF)(V_o)(V_i)}$$

where,

A_x = area of the compound quantitation ion

A_{is} = area of IS quantitation ion

I_s = IS amount in nanograms

RRF = average RRF from curve

V_o = volume of water extracted in ml's

V_i = volume of extract injected in ul's

V_t = volume of the concentrated extract in ul's

D_f = Dilution factor. The dilution factor for the analysis of water samples for semi-volatiles organics by this method is defined as follows:

$$\frac{\text{uL most conc. extract used to make dilution} + \text{uL clean solvent}}{\text{uL most conc. extract used to make dilution}}$$

If no dilution is performed, D_f = 1.0.

Soil Samples

$$\text{Concentration ug/Kg (dry weight basis)} = \frac{(A_x)(I_s)(V_t)(D_f)(2.0)}{(A_{is})(RRF)(V_i)(W_s)(D)}$$

where,

A_x, I_s, A_{is}, V_t, V_i, D_f, and RRF are as given for water.

$$D = \frac{100 - \% \text{ moisture}}{100}$$

W_s = weight of sample extracted in grams

Effective Date: 10/15/01

9.10 Tentatively Identified Compounds

- 9.10.1 An estimated concentration for non-target compounds tentatively identified in the sample shall be determined by the internal standard method. For quantitation, the nearest IS free of interferences shall be used.
- 9.10.2 The equation for calculating concentrations are the same as in 9.9.3. Total area counts from the total ion chromatograms are used for both the IS and compound. A RRF of 1.0 is assumed and the resulting concentration shall be qualified as "J" (estimated) and "N"(presumptive evidence of presence), indicating the quantitative and qualitative uncertainties associated with this non-target compound.
- 9.10.3 If the on-column concentration of any compound in any sample extract exceeds the initial calibration range, that sample extract must be diluted, the IS concentration readjusted, and the sample extract reanalyzed. Guidance in performing dilutions, and exceptions to this requirement are as follows:
- 9.10.3.1 Use the results of the original analysis to determine the approximate dilution factor required to get the largest analyte peak within the initial calibration range.
 - 9.10.3.2 The dilution factor chosen shall keep the response of the largest analyte peak for a target compound in the upper half of the initial calibration range of the instrument.
 - 9.10.3.3 Data for more than two analyses shall not be submitted.
 - 9.10.3.4 Do not dilute MS/MSD samples to get either spiked or non-spiked analytes within the calibration range. If the sample from which the MS/MSD aliquots were taken contains high levels of the spiked analytes, run the MS/MSD at the same dilution as the unspiked sample.

9.11 Instrument Maintenance

- 9.11.1 All instrumentation is covered by a service contract with an external instrumentation service vendor, or by STL personnel trained in preventative maintenance.

Effective Date: 10/15/01

9.11.2 Preventative Maintenance

- 9.11.2.1 Preventative maintenance is performed at routine intervals. All instrument preventative maintenance is performed according the manufacturers recommended procedures, by trained personnel. All preventative maintenance shall be thoroughly documented in the maintenance log, as to a description of the maintenance performed, the date performed, and the personnel performing the maintenance.

9.11.3 Corrective Maintenance Determinants and Procedures

- 9.11.3.1 Corrective maintenance is deemed necessary when the analytical system, after reanalysis, can not meet tune, calibration, or other protocol specific QC criteria. Corrective maintenance may include, but is not limited to, decontamination of the system, source cleaning, replacing the electron multiplier, column replacement or filament replacement. All corrective maintenance is performed according the manufacturers recommended procedures, by trained personnel. All corrective maintenance shall be thoroughly documented in the maintenance log, as to a description of the maintenance performed, the date performed, and the personnel performing the maintenance.

9.11.4 Maintenance Authorization

- 9.11.4.1 All preventative and corrective maintenance is authorized by the laboratory manager, or designee. When maintenance is deemed necessary, a service call is placed for all equipment covered under a service contract, by the department's manager, or designee.

10.0 QUALITY CONTROL

10.1 Initial Demonstration of Precision and Accuracy

- 10.1.1 Prepare four aliquots of the QC Check Standard in reagent water. Process the samples through the whole analytical procedure.
- 10.1.2 Calculate the average recovery (x) in ug/L and the standard deviation (s) in ug/L for each parameter from the four results. Compare the s and x with the criteria listed in Table 4.0. If all parameters meet the acceptance criteria, analysis of samples can begin. If any parameter fails, the cause for the failure must be determined and the test must be repeated for that parameter.

Effective Date: 10/15/01

- 10.1.3 The demonstration of precision and accuracy will be verified on an annual basis.
- 10.2 Method detection limits (MDL's) for this method will be verified on an annual basis as detailed in the latest version of the SOP on MDL's.
- 10.3 Daily Performance Tests
 - 10.3.1 Prior to initiating any data collection activities it is necessary to establish that a given GC/MS system meets the instrument performance criteria. This is accomplished through the analysis of 50 ng or less of decafluorotriphenylphosphine (DFTPP).
 - 10.3.1.1 DFTPP must be analyzed at the start of every 12 hour sequence and can be a component in the 50 ng/uL (100 total ng/2uL) calibration standard.
 - 10.3.1.2 The key ions produced during the analysis of DFTPP and their respective ion abundance criteria are given in Table 3.0. This criteria must be met before any calibration standards, blanks, or samples may be analyzed.
 - 10.3.1.3 If the criteria is not met, the DFTPP must be reanalyzed. Repeated failure shall require the instrument to be manually tuned. After manual tuning, the DFTPP must be reinjected and the abundance criteria must be met before proceeding.
 - 10.3.2 The 12 hour time period for a GC/MS system instrument performance check and standards calibration (initial or continuing calibration criteria) begins at the moment of injection of the DFTPP analysis that is submitted as documentation of a compliant instrument performance check. The time period ends after 12 hours have elapsed according to the system clock.
 - 10.3.3 After the instrument performance criteria is met, the initial calibration curve must be verified through the analysis of a continuing calibration at 50 ng/uL (100 total ng/2uL). The continuing calibration criteria must be met before any method blank or sample analyses may proceed.

Effective Date: 10/15/01

10.3.4 A method blank spiked with surrogates is extracted with every batch of samples and must be analyzed with the sample extracts after calibration criteria has been met. An acceptable method blank must meet the following:

- 10.3.4.1 Less than or equal to 5X the PQL for the phthalate esters.
- 10.3.4.2 Less than or equal to the PQL for each of the other target compounds.
- 10.3.4.3 If the method blank exceeds the above criteria, the analytical system is considered to be out of control. The source of the contamination must be investigated and appropriate corrective measures must be taken and documented before further sample analysis proceeds. All samples analyzed with a method blank that is out of control must be re-extracted and reanalyzed. The problems and solutions must be addressed in the SDG narrative.

10.4 Matrix Spike, Matrix Spike Duplicates and Matrix Spike Blanks

- 10.4.1 An MS and MSD must be extracted and analyzed for each group of samples of a similar matrix within each case, 20 samples, group of samples of a similar concentration level (soils only), or each 7 calendar day period; whichever is more frequent.
- 10.4.2 The limits for matrix spike compound recovery and relative percent difference (RPD) are given in Table 2.0. These limits are only advisory; therefore, no further action is required if the criteria limits are not achieved. However, frequent failures shall be investigated for possible laboratory generated error. The NYSDEC MSB has mandatory acceptance criteria. The MSB is evaluated against the Water % Recoveries listed in Table 2.0. If the MSB does not meet criteria, a QCDF is written, and the project manager is informed.

10.5 QC Check Samples

- 10.5.1 A QC Check Sample must be extracted with each extraction preparation batch for each group of samples of a similar matrix and concentration level (soils only). The QC Check Sample is analyzed with the associated batch of samples.
- 10.5.2 The limits for the spike compound recoveries are given in Table 9.0. These limits are only advisory; therefore no further action is required if the criteria limits are not achieved. However, frequent failures shall be investigated for possible laboratory generated error.

Effective Date: 10/15/01

10.6 Surrogates

- 10.6.1 The surrogates Phenol-d5, 2,4,6-Tribromophenol, 2-Fluorophenol, Nitrobenzene-d5, Terphenyl-d14, and 2-Fluorobiphenyl are added to each sample, blank, standard, and MS/MSD, prior to extraction. The acid surrogates are added at 100 ug and the base-neutrals at 50 ug for waters and soils.
- 10.6.2 Surrogate recoveries must be within the QC limits given in Table 3.0 which were determined from multi-laboratory data. If the recovery for any two acid surrogates (phenol-d5, 2-fluorophenol, and 2,4,6-tribromophenol) or BN surrogates (nitrobenzene-d5, 2-fluorobiphenyl, and terphenyl-d14) are not within the QC limits defined in Table 2.0, or if any one of these surrogate recoveries are below 10%, the following actions are required:
- 10.6.2.1 Check all calculations for accuracy, spiking solutions, and internal standards
 - 10.6.2.2 Reanalyze the sample if none of the above steps reveal a problem
 - 10.6.2.3 Do not reanalyze dilutions if surrogate recoveries are outside limits
 - 10.6.2.4 There is no need to reanalyze the MS or MSD, even if the surrogate recoveries are outside the limits
 - 10.6.2.5 If the sample associated with the MS/MSD does not meet specifications, it should be reanalyzed only if the MS/MSD surrogate recoveries are within the limits
- 10.6.3 If the reanalysis of the sample solves the problem, then only submit the second analysis. If the reanalysis does not solve the problem, then re-extract and reanalyze the sample.
- 10.6.4 If the re-extraction and reanalysis of the sample solves the problem, contact the client by a QCDF and find out what they want submitted. If the re-extraction and reanalysis does not solve the problem, then both sets of results may be reported. Re-extraction and reanalysis shall be decided on a project specific basis and may not be requested by the client.
- 10.6.5 If the recovery of the surrogates in a method blank are not acceptable, as defined in section 10.6.2 above, then the method blank and all associated samples must be re-extracted and reanalyzed.

Effective Date: 10/15/01

10.7 Internal Standards

- 10.7.1 Internal standards are added to each sample, blank, standard, and MS/MSD, at 40 ng prior to injection. Injection of 2 uL of the extract will result in a 80 ng on-column injection of all the internal standards.
- 10.7.2 The retention times (RT) and extracted ion current profile (EICP) of each IS must be evaluated for all standards immediately after the data acquisition. The IS EICP areas must be monitored and evaluated for each sample, blank, MS, MSD. If the IS EICP changes by more than a factor of 2 (-50% to +100%) from the latest (12 hour) calibration standard, the mass spectrometric system must be inspected for malfunctions, and corrections made as required. If the RT for any IS changes by more than 30 seconds from the latest (12 hour) calibration standard, the chromatographic system must be inspected for malfunctions, and corrections made as required. When corrections are made, reanalysis of the samples analyzed while the system was malfunctioning is necessary. If no system malfunctioning occurred, then document the matrix interference problem via a corrective action report. If the sample associated with the MS/MSD does not meet specifications, it should be reanalyzed only if the MS/MSD IS criteria is within limits.
- 10.7.3 If the reanalysis of the sample solves the problem, then only submit the second analysis.

10.8 Manual Integrations and Editing Flags

- 10.8.1 Manual integrations are required when the automated software does not correctly integrate extracted ion current profiles (EICP). Manual integrations are flagged by the data system with the "M" qualifier beside any manually integrated area on the hardcopy quant report. The analyst shall initial and date the hardcopy report for the manual integrations performed.

10.9 Analytical Documentation Procedures

10.9.1 Instrument Batches

- 10.9.2 An instrument batch is created for each analytical sequence to organize all the associated data. Batch designations are of the format:

MMDDYY-X

where X = instrument identifier
MMDDYY = Month, Day, Year
(i.e. 021500-1)

Effective Date: 10/15/01

10.9.3 Instrument batches are number sequentially so each analytical sequence is identified by a unique batch identifier. The batch consists of a file folder with all the associated QC information for the analytical sequence. The raw data is then bound together with the file folder to complete the batch.

10.10 Filing System

10.10.1 All active batches are filed chronologically according to instrument. The batches are transferred to file boxes for long term storage once all the associated data within a batch has been completed.

10.11 Data Archiving

10.11.1 The data files and method files on the server are archived on a daily basis.

10.12 Instrument Run Logs

10.12.1 It is STL's policy that all measurement data be recorded in logbooks or on preprinted log sheets in permanent ink. Transcriptions shall be avoided whenever possible. The record shall reflect the measurement performed and all appropriate details for conclusions related to the measurement. The record shall be signed and dated by the individual performing the measurement on the day the measurement is performed. Corrections shall be made by drawing a single line through the error, and initialing and dating the correction. A secondary authorization of the logbook is required and shall be performed by the department's manager or designee.

10.12.2 Each instrument has its own set of bound run logs which are sequentially number and paginated. Run logs are filed in the laboratory once they have been filled, for future reference. Each analytical sequence shall be started on a new page of the log and continued on the next page, if necessary. The header information designating the standard codes used shall be completed for each sequence. All standards used are recorded in this field for future traceability. The data file, job number, sample number, quantitation factor, dilution factor, analyst's signature, and date are recorded.

Effective Date: 10/15/01

10.13 Initial data review sheet

10.13.1 The initial data review sheet (IDRS) is a computerized review sheet which is used to check the key quality control criteria for compliance. The IDRS is used to check that all samples have been analyzed with the required calibration time frame, if the IS's meet RT and EICP area criteria, and if the ID file being utilized was correctly updated. The IDRS is also used as the initial data review tool. Each sample is listed on the sheet and it is either accepted or rejected in the right hand column, by the analyst performing the data review. If reruns are required for dilutions, then the analyst shall indicate the proper dilution required for reanalysis. The data reviewer initials and dates the IDRS. The batch is then secondary reviewed by another analyst to ensure that no oversight errors were made during the initial review. The batch is then filed for use during deliverables preparation. Refer to Figure 3.0 for an example of the IDRS.

10.14 Quality Control Deviation Form

10.14.1 A quality control deviation form (QCDF) is issued when a problem is encountered during analysis, data reduction or deliverables preparation, data validation, or when any deviations from this SOP occur. The QCDF is prepared by the analyst first identifying the problem and is then submitted to the department's manager for approval. The manager shall submit the QCDF to the QA/QC department where the QCDF will be recorded, copied and distributed to the necessary departments. A copy shall be placed in the job file by the project manager.

10.14.2 The lower portion of the QCDF is for the corrective actions taken, and is completed by the department or project manager when a corrective action decision has been made. The QCDF is then redistributed to all the departments and individuals involved.

10.15 Chain of Custody Record

10.15.1 When samples are removed from storage for preparation or analysis they must be signed out utilizing the chain of custody record (COC). The samples shall then be signed back in on the COC upon their return to storage or designated "used" if the sample volume is consumed during the preparation or analysis.

Effective Date: 10/15/01

10.16 Sample Extraction Log

10.16.1 Samples are tracked on the Semivolatile Sample Extraction Log. As the extraction is being completed, the extraction information is entered into the extraction log. Samples requiring reanalysis are also tracked with the reason for the reanalysis (i.e. dilution, I.S. confirmation, etc). If a re-extraction is required, a QCDF is issued.

11.0 CALCULATIONS

11.1 Relative Response Factor (RRF)

$$\text{RRF} = \frac{(A_x)(C_{is})}{(A_{is})(C_x)}$$

where,

A_x = area of the compound quantitation ion

A_{is} = area of IS quantitation ion

C_{is} = IS concentration

C_x = compound concentration

An average RRF is calculated for each compound and surrogate from the initial calibration.

11.2 Percent Relative Standard Deviation (%RSD)

$$\%RSD = \frac{\text{Standard Deviation}}{\text{mean}} \times 100$$

11.3 Percent Difference (%D)

$$\%D = \frac{(\text{Average RRF}_i) - (\text{RRF}_c)}{(\text{Average RRF}_i)} \times 100$$

where,

Average RRF_i = Average RRF from the initial calibration

RRF_c = RRF from the continuing calibration standard

Effective Date: 10/15/01

11.4 Percent Moisture

$$\% \text{ moisture} = \frac{\text{g of Wet Sample} - \text{g of Dry Sample}}{\text{g of Wet Sample}} \times 100$$

11.5 Target Compound Concentrations

The calculations used to determine the target compound concentrations are described in the sections above.

11.6 Surrogate Percent Recovery

$$\% \text{ Recovery} = \frac{\text{Concentration Found}}{\text{Concentration Spiked}} \times 100$$

11.7 Matrix Spike Recovery/Matrix Spike Duplicate Recovery/Full Matrix Spike Recovery

$$\% \text{ Recovery} = \frac{\text{SSR} - \text{SR}}{\text{SA}} \times 100$$

where,

SSR = spiked sample result

SR = sample result

SA = spike added

11.8 Relative Percent Difference

$$\text{RPD} = \frac{\text{Absolute (MSR - MSDR)}}{(1/2)(\text{MSR} + \text{MSDR})} \times 100$$

where,

MSR = matrix spike recovery

MSDR = matrix spike duplicate recovery

The absolute value of the recovery difference is used in the above equation.

Effective Date: 10/15/01

11.9 Adjusted Estimated Quantitation Limit for Samples

$$\text{Adjusted EQL} = \frac{(\text{EQL}) \times \text{Df}}{\text{D}}$$

where,

$$\text{D} = \frac{100 - \% \text{ Moisture}}{100}$$

$$\text{Df} = \text{Dilution Factor}$$

12.0 DATA REDUCTION and REVIEW

- 12.1 Hardcopies of all instrument calibration, calibration verification and daily analytical sequence raw data shall be assembled into “packages” for review.
- 12.2 Analysts are responsible for reviewing the packages to evaluate the accuracy of transcriptions, the instrument’s performance, and the calculated results. Section 10 details the acceptance criteria and corrective actions for instrument and batch QA/QC.
- 12.3 All necessary documentation to support corrective actions performed shall be included with the packages and available for review.
- 12.4 Preliminary results may be reported to the client at this time.

13.0 REPORTING OF RESULTS

- 13.1 Prior to generating a final report of results, the data is reviewed a second time. In addition to the method review performed by the first chemist, the second review includes a verification of any project/client specific criteria and a sensibility check.
- 13.2 If for any reason there is a question regarding the technical soundness of the data, it is brought directly to the attention of the Technical Director and/or Quality Assurance Manager.
- 13.3 After completing the secondary review, the final results are reported to the client in hardcopy form and/or electronic deliverable, if requested.

Effective Date: 10/15/01

14.0 POLLUTION PREVENTION

- 14.1 Where possible, all STL-OST methods utilize very small quantities of extraction solvents to minimize pollution.
- 14.2 For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction", available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W. Washington D.C. 20036, (202) 872-4477.

15.0 WASTE MANAGEMENT

- 15.1 Solvent waste must be disposed of in clearly labeled waste cans.
- 15.2 Acid waste must be collected in clearly labeled acid waste containers.
- 15.3 Solid materials (gloves, soiled paper products, etc.) are placed in the solid debris container. Do not put liquids in the solid waste container.
- 15.4 Refer to the Laboratory Sample and Waste Disposal plan.
- 15.5 Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of STL. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16.0 SUPPLEMENTAL DOCUMENTS – N/A

17.0 REFERENCES

- 17.1 "Test Methods for Evaluating Solid Waste", USEPA-SW846, Third Edition, September 1986 with all current revisions, method 8270C.

18.0 SUBSTANTIVE REVISIONS

- 18.1 Original issue – 05/15/01
- 18.2 Revision – 10/15/01

Effective Date: 10/15/01

TABLE 1.0
TARGET COMPOUNDS AND REPORTING LEVELS

| Semi-Volatile Organics | Quantitation Limits* | |
|-------------------------------|----------------------|------------------|
| | Water µg/L | Low Soil (µg/Kg) |
| Phenol | 10 | 330 |
| bis(2-Chloroethyl)ether | 10 | 330 |
| 2-Chlorophenol | 10 | 330 |
| 1,2-Dichlorobenzene | 10 | 330 |
| 1,4-Dichlorobenzene | 10 | 330 |
| Benzyl Alcohol | 10 | 330 |
| 1,3-Dichlorobenzene | 10 | 330 |
| 2-Methylphenol | 10 | 330 |
| 2,2'-oxybis(1-Chloropropane)# | 10 | 330 |
| 4-Methylphenol | 10 | 330 |
| N-Nitroso-di-n-propylamine | 10 | 330 |
| Hexachloroethane | 10 | 330 |
| Nitrobenzene | 10 | 330 |
| Isophorone | 10 | 330 |
| 2-Nitrophenol | 10 | 330 |
| 2,4-Dimethylphenol | 10 | 330 |
| Benzoic Acid | 50 | 1600 |
| bis(2-Chloroethoxy)methane | 10 | 330 |
| 2,4-Dichlorophenol | 10 | 330 |
| 1,2,4-Trichlorobenzene | 10 | 330 |
| Naphthalene | 10 | 330 |
| 4-Chloroaniline | 10 | 330 |
| Hexachlorobutadiene | 10 | 330 |
| 4-Chloro-3-methylphenol | 10 | 330 |
| 2-Methylnaphthalene | 10 | 330 |
| Hexachlorocyclopentadiene | 10 | 330 |
| 2,4,6-Trichlorophenol | 25 | 800 |
| 2,4,5-Trichlorophenol | 25 | 800 |
| 2-Chloronaphthalene | 10 | 330 |
| 2-Nitroaniline | 25 | 800 |
| Dimethylphthalate | 10 | 330 |
| Acenaphthylene | 10 | 330 |
| 3-Nitroaniline | 25 | 800 |
| Acenaphthene | 10 | 330 |
| 2,4-Dinitrophenol | 25 | 800 |
| 4-Nitrophenol | 25 | 800 |
| Dibenzofuran | 10 | 330 |
| 2,4-Dinitrotoluene | 10 | 330 |
| Diethylphthalate | 10 | 330 |
| 4-Chlorophenyl-phenylether | 10 | 330 |
| Fluorene | 10 | 330 |
| 4-Nitroaniline | 20 | ND |
| 4,6-Dinitro-2-methylphenol | 25 | 800 |
| N-nitrosodiphenylamine | 10 | 330 |
| 4-Bromophenyl-phenylether | 10 | 330 |
| Hexachlorobenzene | 10 | 330 |
| Pentachlorophenol | 25 | 800 |
| Phenanthrene | 10 | 330 |
| Anthracene | 10 | 330 |
| Di-n-butylphthalate | 10 | ND |
| Fluoranthene | 10 | 330 |
| Pyrene | 10 | 330 |
| Butylbenzylphthalate | 10 | 330 |
| 3,3'-dichlorobenzidine | 10 | 330 |
| Benzo(a)anthracene | 10 | 330 |
| Chrysene | 10 | 330 |
| bis(2-Ethylhexyl)phthalate | 10 | 330 |
| Di-n-octylphthalate | 10 | 330 |
| Benzo(b)fluoranthene | 10 | 330 |
| Benzo(k)fluoranthene | 10 | 330 |
| Benzo(a)pyrene | 10 | 330 |
| Indeno(1,2,3-cd)pyrene | 10 | 330 |
| Dibenzo(a,h)anthracene | 10 | 330 |
| Benzo(g,h,i)perylene | 10 | 330 |

*Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

#Previously known as bis(2-chloroisopropyl)ether.

Effective Date: 10/15/01

TABLE 2.0
MATRIX SPIKE RECOVERY AND RELATIVE PERCENT DIFFERENCE LIMITS

| Compound | % Recovery Water | RPD Water | % Recovery Soil | RPD Soil |
|----------------------------|---------------------|--------------|--------------------|-------------|
| Phenol | 12-110 | 42 | 26-90 | 35 |
| 2-Chlorophenol | 27-123 | 40 | 25-102 | 50 |
| 1,4-Dichlorobenzene | 36-97 | 28 | 28-104 | 27 |
| N-Nitroso-di-n-propylamine | 41-116 | 38 | 41-126 | 38 |
| 1,2,4-Trichlorobenzene | 39-98 | 28 | 38-107 | 23 |
| 4-Chloro-3-methylphenol | 23-97 | 42 | 26-103 | 33 |
| Acenaphthene | 46-118 | 31 | 31-137 | 19 |
| 4-Nitrophenol | 10-80 | 50 | 11-114 | 50 |
| 2,4-Dinitrotoluene | 24-96 | 38 | 28-89 | 47 |
| Pentachlorophenol | 9-103 | 50 | 17-109 | 47 |
| Pyrene | 26-127 | 31 | 35-142 | 36 |

Effective Date: 10/15/01

TABLE 3.0
SURROGATE RECOVERY LIMITS

| Compound | % Recovery Water | % Recovery Soil |
|-----------------------------|-----------------------------|----------------------------|
| Nitrobenzene-d ₅ | 35-114 | 23-120 |
| 2-Fluorobiphenyl | 43-116 | 30-115 |
| Terphenyl-d ₁₄ | 33-141 | 18-137 |
| Phenol-d ₅ | 10-110 | 24-113 |
| 2-Fluorophenol | 21-110 | 25-121 |
| 2,4,6-Tribromophenol | 10-123 | 19-122 |

Effective Date: 10/15/01

TABLE 4.0
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA FOR
QUADRAPOLE MASS SPECTROMETERS

| Mass | Ion Abundance Criteria |
|------|--|
| 51 | 30.0 - 60 percent of mass 198 |
| 68 | Less than 2.0 percent of mass 69 |
| 69 | Present |
| 70 | Less than 2.0 percent of mass 69 |
| 127 | 40-60 percent of mass 198 |
| 197 | Less than 1.0 percent of mass 198 |
| 198 | Base peak, 100 percent relative abundance (see note) |
| 199 | 5.0-9.0 percent of mass 198 |
| 275 | 10.0 - 30.0 percent of mass 198 |
| 365 | Greater than 1 percent of mass 198 |
| 441 | Present, but less than mass 443 |
| 442 | 40.0 - 100.0 percent of mass 198 |
| 443 | 17 - 23 percent of mass 442 |

Effective Date: 10/15/01

TABLE 5.0

| Calibration Check Compounds (ccc) | |
|--|------------------------|
| Phenol | Acenaphthene |
| 1,4-Dichlorobenzene | N-Nitrosodiphenylamine |
| 2-Nitrophenol | Pentachlorophenol |
| 2,4-Dichlorophenol | Fluoranthene |
| Hexachlorobutadiene | Di-n-octylphthalate |
| 4-Chloro-3-methylphenol | Benzo(a)pyrene |
| 2,4,6-Trichlorophenol | |

TABLE 5.1

| System Performance Check Compounds (spcc) | |
|--|---------------------------|
| N-Nitroso-di-n-propylamine | Hexachlorocyclopentadiene |
| 2,4-Dinitrophenol | 4-Nitrophenol |

Effective Date: 10/15/01

**TABLE 6.0 - SEMI-VOLATILE INTERNAL STANDARDS WITH CORRESPONDING TARGET COMPOUNDS
AND SURROGATES ASSIGNED FOR QUANTITATION**

| 1,4-Dichlorobenzene-d ₄ | Naphthalene-d ₈ | Acenaphthene-d ₁₀ | Phenanthrene-d ₁₀ | Chrysene-d ₁₂ | Perylene-d ₁₂ |
|------------------------------------|------------------------------------|------------------------------|------------------------------|----------------------------------|--------------------------|
| Phenol | Nitrobenzene | Hexachlorocyclopentadiene | 4,6-Dinitro-2-methylphenol | Pyrene | Di-n-octylphthalate |
| bis(2-Chloroethyl)ether | Isophorone | 2,4,6-Trichlorophenol | N-Nitrosodiphenylamine | Butylbenzylphthalate | Benzo(b)fluoranthene |
| 2-Chlorophenol | 2-Nitrophenol | 2-Chloronaphthalene* | 4-Bromophenyl phenyl ether | 3,3'-Dichlorobenzidine | Benzo(k)fluoranthene |
| 1,3-Dichlorobenzene | 2,4-Dimethyl-phenol | 2-Nitroaniline | Hexachlorobenzene | Benzo(a)anthracene | Benzo(a)pyrene |
| 1,4-Dichlorobenzene | bis(2-Chloroethoxy)methane | Dimethylphthalate | Pentachlorophenol | bis(2-Ethylhexyl)phthalate | Indeno(1,2,3-cd)pyrene |
| 1,2-Dichlorobenzene | 2,4-Dichlorophenol | Acenaphthylene | Phenanthrene | Chrysene | Dibenz(a,h)anthracene |
| 2-Methylphenol | 1,2,4-Trichlorobenzene | 3-Nitroaniline | Anthracene | Terphenyl-d ₁₄ (surr) | Benzo(g,h,i)perylene |
| 2,2'-oxybis-(1-Chloropropane) | Naphthalene | Acenaphthene | Di-n-butylphthalate | | |
| 4-Methylphenol | 4-Chloroaniline | 2,4-Dinitrophenol | Fluoranthene | | |
| N-Nitroso-di-n-propylamine | Hexachlorobutadiene | 4-Nitrophenol | | | |
| Hexachloroethane | 4-Chloro-3-methylphenol | Dibenzofuran | | | |
| 2-Fluorophenol (surr) | 2-Methylnaphthalene | 2,4-Dinitrotoluene | | | |
| Phenol-d ₅ (surr) | Nitrobenzene-d ₅ (surr) | 2,6-Dinitrotoluene | | | |
| | | Diethylphthalate | | | |
| | | 4-Chlorophenyl phenyl ether | | | |
| | | Fluorene | | | |
| | | 4-Nitroaniline | | | |
| | | 2-Fluorobiphenyl (surr) | | | |
| | | 2,4,6-Tribromophenol (surr) | | | |

Effective Date: 10/15/01

TABLE 7.0
CHARACTERISTIC IONS FOR INTERNAL STANDARDS
FOR SEMI-VOLATILE COMPOUNDS

| Internal Standards | Primary Ion | Secondary Ions |
|------------------------------------|-------------|----------------|
| 1,4-Dichlorobenzene-d ₄ | 152 | 115 |
| Naphthalene-d ₈ | 136 | 68 |
| Acenaphthene-d ₁₀ | 164 | 162, 160 |
| Phenanthrene-d ₁₀ | 188 | 94, 80 |
| Chrysene-d ₁₂ | 240 | 120, 236 |
| Perylene-d ₁₂ | 264 | 260, 265 |

Effective Date: 10/15/01

TABLE 8.0
CHARACTERISTIC IONS FOR TARGET COMPOUNDS, INTERNAL STANDARDS AND SYSTEM MONITORING COMPOUNDS

| Parameter | Primary Ion | Secondary Ion(s) |
|-------------------------------|-------------|------------------|
| Phenol | 94 | 65, 66 |
| bis(2-Chloroethyl)ether | 93 | 63, 95 |
| 2-Chlorophenol | 128 | 64, 130 |
| 1,3-Dichlorobenzene | 146 | 148, 113 |
| 1,4-Dichlorobenzene | 146 | 148, 113 |
| 1,3-Dichlorobenzene | 146 | 148, 113 |
| 2-Methylphenol | 108 | 107 |
| 2,2'-oxybis(1-Chloropropane)# | 45 | 77, 79 |
| 4-Methylphenol | 108 | 107 |
| N-Nitroso-d,n-propylamine | 70 | 42, 101, 130 |
| Hexachloroethane | 117 | 201, 199 |
| Nitrobenzene | 77 | 123, 65 |
| Isophorone | 82 | 95, 138 |
| 2-Nitrophenol | 139 | 65, 109 |
| 2,4-Dimethylphenol | 107 | 121, 122 |
| bis(2-Chloroethoxymethane | 93 | 95, 123 |
| 2,4-Dichlorophenol | 162 | 164, 98 |
| 1,2,4-Trichlorobenzene | 180 | 182, 145 |
| Naphthalene | 128 | 129, 127 |
| 4-Chloroaniline | 127 | 129 |
| Hexachlorobutadiene | 225 | 223, 227 |
| 4-Chloro-3-methylphenol | 107 | 144, 142 |
| 2-Methylnaphthalene | 142 | 141 |
| Hexachlorocyclopentadiene | 237 | 235, 272 |
| 2,4,6-Trichlorophenol | 196 | 198, 200 |
| 2,4,5-Trichlorophenol | 196 | 198, 200 |
| 2-Chloronaphthalene | 162 | 164, 127 |
| 2-Nitroaniline | 65 | 92, 138 |
| Dimethylphthalate | 163 | 194, 164 |
| Acenaphthylene | 152 | 151, 153 |
| 3-Nitroaniline | 133 | 108, 92 |
| Acenaphthene | 153 | 152, 154 |
| 2,4-Dinitrophenol | 184 | 63, 154 |
| 4-Nitrophenol | 109 | 139, 65 |
| Dibenzofuran | 168 | 139 |
| 2,4-Dinitrotoluene | 165 | 63, 182 |
| 2,6-Dinitrotoluene | 165 | 39, 121 |
| Diethylphthalate | 149 | 177, 150 |
| 4-Chlorophenyl-phenylether | 204 | 206, 141 |
| Fluorene | 166 | 165, 167 |
| 4-Nitroaniline | 138 | 92, 108 |
| 4,6-Dinitro-2-methylphenol | 198 | 182, 77 |
| N-nitrosodiphenylamine | 169 | 168, 167 |
| 4-Bromophenyl-phenylether | 248 | 250, 141 |
| Hexachlorobenzene | 284 | 142, 249 |
| Pentachlorophenol | 266 | 264, 268 |
| Phenanthrene | 178 | 179, 176 |
| Anthracene | 178 | 179, 176 |
| Di-n-butylphthalate | 149 | 150, 104 |
| Fluoranthene | 202 | 101, 100 |
| Pyrene | 202 | 101, 100 |
| Butylbenzylphthalate | 149 | 91, 206 |
| 3,3-dichlorobenzidine | 252 | 254, 126 |
| Benzo(a)anthracene | 228 | 229, 226 |
| bis(2-Ethylhexyl)phthalate | 149 | 167, 279 |
| Chrysene | 228 | 226, 229 |
| Di-n-octylphthalate | 149 | --- |
| Benzo(b)fluoranthene | 252 | 253, 125 |
| Benzo(k)fluoranthene | 252 | 253, 125 |
| Benzo(a)pyrene | 252 | 253, 125 |
| Indeno(1,2,3-cd)pyrene | 276 | 138, 227 |
| Dibenzof(a,h)anthracene | 278 | 139, 279 |
| Benzo(g,h,i)perylene | 276 | 138, 277 |

SURROGATES

| Parameter | Primary Ion | Secondary Ion(s) |
|----------------------|-------------|------------------|
| Phenol-d5 | 99 | 42, 71 |
| 2-Fluorophenol | 112 | 64 |
| 2,4,6-Tribromophenol | 330 | 332, 141 |
| Nitrobenzene-d5 | 82 | 128, 54 |
| 2-Fluorobiphenyl | 172 | 171 |
| Terphenyl | 244 | 122, 212 |

Effective Date: 10/15/01

TABLE 2.0 - QC ACCEPTANCE CRITERIA - METHOD 8270

| Semi-Volatile Organics | Limit for s (ug/l) | Range for X (ug/l) | Range for % Recovery |
|-------------------------------|--------------------|--------------------|----------------------|
| Phenol | 22.6 | 8.8 - 50 | 5 - 112 |
| Bis(2-Chloroethyl)ether | 55.0 | 21.4 - 63 | 12 - 158 |
| 2-Chlorophenol | 8.7 | 18.1 - 60.2 | 23 - 134 |
| 1,2-Dichlorobenzene | 30.9 | 4.3 - 56 | 32 - 129 |
| 1,4-Dichlorobenzene | 32.1 | 18.6 - 52.8 | 20 - 124 |
| Benzidine | N.L. | N.L. | N.L. |
| 1,3-Dichlorobenzene | 41.7 | 8.35 - 76.9 | D - 172 |
| 2,6-Dinitrotoluene | 29.6 | 34 - 68.3 | 50 - 158 |
| 2,2'-oxybis(1-Chloropropane)# | 46.3 | 31.4 - 69.3 | 36 - 166 |
| N-nitrosodimethylamine | N.L. | N.L. | N.L. |
| N-Nitroso-di-n-propylamine | 100 | 6.8 - 98.9 | D - 230 |
| Hexachloroethane | 24.5 | 27.6 - 50 | 40 - 113 |
| Nitrobenzene | 39.3 | 27.1 - 78.8 | 35 - 180 |
| Isophorone | 63.3 | 23.3 - 90.1 | 21 - 196 |
| 2-Nitrophenol | 35.2 | 22.5 - 83.3 | 29 - 182 |
| 2,4-Dimethylphenol | 26.4 | 20.9 - 54.5 | 32 - 119 |
| bis(2-Chloroethoxy)methane | 34.5 | 24.6 - 82.3 | 33 - 184 |
| 2,4-Dichlorophenol | 26.4 | 26.5 - 60.8 | 39 - 135 |
| 1,2,4-Trichlorobenzene | 28.1 | 28.6 - 64.6 | 44 - 142 |
| Naphthalene | 30.1 | 17.8 - 59.8 | 21 - 133 |
| Hexachlorobutadiene | 26.3 | 18.9 - 51.1 | 24 - 116 |
| 4-Chloro-3-methylphenol | 37.2 | 20.4 - 63.9 | 22 - 147 |
| Hexachlorocyclopentadiene | N.L. | N.L. | N.L. |
| 2,4,6-Trichlorophenol | 31.7 | 26.2 - 64.6 | 37 - 144 |
| 2-Chloronaphthalene | 13.0 | 32.2 - 56.7 | 60 - 118 |
| Dimethylphthalate | 23.2 | D - 50 | D - 112 |
| Acenaphthylene | 40.2 | 26.7 - 63 | 33 - 145 |
| Acenaphthene | 27.6 | 30.0 - 66.1 | 47 - 145 |
| 2,4-Dinitrophenol | 49.8 | D - 86.4 | D - 191 |
| 4-Nitrophenol | 47.2 | 6.5 - 53.2 | D - 132 |

Effective Date: 10/15/01

TABLE 9.0 (continued)

| Semi-Volatile Organics | Limits for s (ug/l) | Range for X, (ug/l) | Limits for % Recovery |
|----------------------------|---------------------|---------------------|-----------------------|
| 2,4-Dinitrotoluene | 21.8 | 23.7 - 63.4 | 39 - 139 |
| Diethylphthalate | 26.5 | D - 50 | D - 114 |
| 4-Chlorophenyl-phenylether | 33.4 | 19.2 - 72.3 | 25 - 158 |
| Fluorene | 20.7 | 35.8 - 53.2 | 59 - 121 |
| 4,6-Dinitro-2-methylphenol | 93.2 | 26.5 - 50 | D - 181 |
| N-nitrosodiphenylamine | N.L. | N.L. | N.L. |
| 4-Bromophenyl-phenylether | 23.0 | 32.4 - 57.2 | 53 - 127 |
| Hexachlorobenzene | 24.9 | 3.9 - 70.7 | D - 152 |
| Pentachlorophenol | 48.9 | 19.0 - 75.9 | 14 - 176 |
| Phenanthrene | 20.6 | 32.6 - 54.3 | 54 - 120 |
| Anthracene | 32.0 | 21.7 - 59.0 | 27 - 133 |
| Di-n-butylphthalate | 16.7 | 4.2 - 55.5 | 1 - 118 |
| Fluoranthene | 32.8 | 21.4 - 60.6 | 26 - 137 |
| Pyrene | 25.2 | 34.8 - 50 | 52 - 115 |
| Butylbenzylphthalate | 23.4 | D - 69.9 | D - 152 |
| 3,3'-Dichlorobenzidine | 71.4 | 4.1 - 106.2 | D - 262 |
| Benzo(a)anthracene | 27.6 | 20.9 - 66.5 | 33 - 143 |
| Chrysene | 48.3 | 22.0 - 69.9 | 17 - 168 |
| bis(2-Ethylhexyl)phthalate | 41.1 | 14.4 - 68.4 | 8 - 158 |
| Di-n-octylphthalate | 31.4 | 9.3 - 65.9 | 4 - 146 |
| Benzo(b)fluoranthene | 38.8 | 21.0 - 70.2 | 24 - 159 |
| Benzo(k)fluoranthene | 32.3 | 12.6 - 72.8 | 11 - 162 |
| Benzo(a)pyrene | 39.0 | 15.8 - 74.0 | 17 - 163 |
| Indeno(1,2,3-cd)pyrene | 44.6 | D - 75.4 | D - 171 |
| Dibenzo(a,h)anthracene | 70.0 | D - 99.8 | D - 227 |
| Benzo(g,h,i)perylene | 58.9 | D - 97.5 | D - 219 |

#Previously known as bis(2-chloroisopropyl)ether.
All test concentrations at 20 ug/l.